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L4: Entry 2 of 5

File: USPT

Feb 18, 2003

DOCUMENT-IDENTIFIER: US 6520425 B1

TITLE: Process and apparatus for the production of nanofibers

Brief Summary Text (4):

It is known to produce nanofibers by using <u>electrospinning</u> techniques. These techniques, however, have been problematic because some spinnable fluids are very viscous and require higher forces than electric fields can supply before sparking occurs, i.e., there is a dielectric breakdown in the air. Likewise, these techniques have been problematic where higher temperatures are required because high temperatures increase the conductivity of structural parts and complicate the control of high electrical fields.

Brief Summary Text (5):

It is known to use pressurized gas to create polymer fibers by using melt-blowing techniques. According to these techniques, a stream of molten polymer is extruded into a jet of gas. These polymer fibers, however, are rather large in that the fibers are greater than 1,000 nanometers (1 micron) in diameter and more typically greater than 10,000 nanometers (10 microns) in diameter. It is also known to combine electrospinning techniques with melt-blowing techniques. But, the combination of an electric field has not proved to be successful in producing nanofibers inasmuch as an electric field does not produce stretching forces large enough to draw the fibers because the electric fields are limited by the dielectric breakdown strength of air.

Detailed Description Text (15):

According to this embodiment, nanofibers are produced by using the apparatus of FIG. 6 according to the following method. Pressure is applied to the container so that fiber-forming material is forced from storage space 35 into gas jet space 14. The pressure that is applied can result from gas pressure, pressurized fluid, or molten polymer from an extruder. Simultaneously, pressurized gas is forced from a gas source 18, through center tube 11, and exits through center tube orifice 15 into gas jet space 14. As with previous embodiments, heat may be applied to the fiber-forming material prior to or after being placed in fiber-forming material container 34, to the pressurized gas entering center tube 11, and/or to storage space 35 by heat source 39 or additional heat sources. Fiber-forming material exiting from storage space 35 into gas jet space 14 forms a thin layer of fiber-forming material on the inside wall of gas jet space 14. This layer of fiber-forming material is subjected to shearing deformation, or other modes of deformation such as surface wave, by the gas jet until it reaches container outlet orifice 36. There the layer of fiber-forming material is blown apart, into many small strands, by the expanding gas.

Detailed Description Text (22):

As with previous embodiments, the outer gas tube 19 extends along supply tube 12 and thereby creates an annular column of gas 21. The lower end 22 of gas annular column 21 and the lower end 23 of supply tube 12 form a lip cleaner orifice 20. In this embodiment, lower end 22 and lower end 23 are on the same horizontal plane (flush) a shown in FIG. 7. As noted above, however, lower ends 22 and 23 may be on different horizontal planes. The pressurized of gas exiting through lip cleaner orifice 20 prevents the buildup of residual amounts of fiber-forming material that can accumulate at lower end 23 of supply tube 12. Simultaneously, pressurized gas is supplied by gas source 28 through shroud gas inlet tube 54 to shroud gas tube 31.

Pressurized gas is forced through the shroud gas tube 31 and it exits from the shroud gas tube orifice 32 thereby creating a shroud of gas around the nanofibers that control the cooling rate of the nanofibers exiting from tube orifice 16. In one particular embodiment, fiber-forming material is supplied by an extruder.

Detailed Description Text (39):

In another embodiment, NGJ is combined with electrospinning techniques. In these combined process, NGJ improves the production rate while the electric field maintains the optimal tension in the jet to produce orientation and avoid the appearance of beads on the fibers. The electric field also provides a way to direct the nanofibers along a desired trajectory through processing machinery, heating ovens, or to a particular position on a collector. Electrical charge on the fiber can also produce looped and coiled nanofibers that can increase the bulk of the non-woven fabric made from these nanofibers.

Detailed Description Text (42):

It should also be appreciated that the average diameter and the range of diameters is affected by adjusting the gas temperature, the flow rate of the gas stream, the temperature of the fluid, and the flow rate of fluid. The flow of the fluid can be controlled by a valve arrangement, by an extruder, or by separate control of the pressure in the container and in the center tube, depending on the particular apparatus used.

 $\frac{\text{Detailed Description Text}}{\text{It should thus be evident that the NGJ methods and apparatus disclosed herein are}}$ capable of providing nanofibers by creating a thin layer of fiber-forming material on the inside of an outlet tube, and this layer is subjected to shearing deformation until it reaches the outlet orifice of the tube. There, the layer of fiber-forming material is blown apart, into many small jets, by the expanding gas. No apparatus has ever been used to make nanofibers by using pressurized gas. Further, the NGJ process creates fibers from spinnable fluids, such as mesophase pitch, that can be converted into high strength, high modulus, high thermal conductivity graphite fibers. It can also produce nanofibers from a solution or melt. It may also lead to an improved nozzle for production of small droplets of liquids. It should also be evident that NGJ produces nanofibers at a high production rate. NGJ can be used alone or in combination with either or both melt blowing or electrospinning to produce useful mixtures of fiber geometries, diameters and lengths. Also, NGJ can be used in conjunction with an electric field, but it should be appreciated that an electric field is not required.

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1. Document ID: US 6645618 B2

L4: Entry 1 of 5

File: USPT

Nov 11, 2003

US-PAT-NO: 6645618

DOCUMENT-IDENTIFIER: US 6645618 B2

TITLE: Aliphatic polyester microfibers, microfibrillated articles and use thereof

DATE-ISSUED: November 11, 2003

INVENTOR-INFORMATION:

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US-CL-CURRENT: 428/359; 428/395, 428/397, 428/399

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2. Document ID: US 6520425 B1

L4: Entry 2 of 5

File: USPT

Feb 18, 2003

US-PAT-NO: 6520425

DOCUMENT-IDENTIFIER: US 6520425 B1

TITLE: Process and apparatus for the production of nanofibers

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